

Addendum To:
Test Protocol and Results for the Determination of Permeation Rates from High Density Polyethylene Containers & Barrier Surface Treatment Feasibility Study

Introduction

Air Resources Board staff tested several High Density Polyethylene (HDPE) portable fuel containers (containers) to determine average permeation rates and to assess the effectiveness of several barrier surface treatments. During previous tests, issues concerning several of the barrier surface treated containers became obvious when reviewing the data. In question were the performance of two sets of level 5 fluorinated containers and all of the sulfonated containers. After considerable review, staff determined that the suspect containers were incorrectly barrier surface treated. It was determined that the sulfonated containers were barrier surface treated with the wrong type of sulfite gas due to a communications error. Furthermore, two sets of level 5 fluorinated containers were also incorrectly treated, possibly due to a mechanical malfunction at the treatment facility. New containers were purchased and submitted to both facilities for treatment and follow-up permeation tests performed. As in the previous tests, containers were subjected to a variable temperature profile and permeation rates determined gravimetrically. All containers were tested with CERT fuel. In all, 20 portable fuel containers were tested over a period of two weeks. Containers selected for testing were purchased at retail outlets located throughout California. The test containers ranged in size from 2.0625 gallons to 6.6 gallons and all initial tests were performed in duplicate.

Test Protocol

All containers selected for testing were preconditioned with fuel for a period of four weeks, minimum. During the preconditioning cycle containers were stored at ambient temperature and pressure in flammable storage cabinets. After a minimum of four weeks preconditioning, the containers were emptied, blown dry with compressed zero air, and immediately refilled with CERT fuel (see Attachment 1). CERT fuel was selected to minimize variation of the permeation results due to variations in fuel properties.

Each container was then sealed using a combination of metal filled epoxy with an overcoat of a special non-permeable two-part epoxy resin (SealPak CS3204 A1/2 Sealant). Where possible, plastic caps and plugs were removed from the containers and replaced with metal plugs and caps. All secondary vents were tapped and plugged with 1/8" brass fittings, band clamped, and coated with sealant.

After allowing sufficient time for the curing of all sealant, the containers were tested for leaks. Containers were heated and when positive pressure was observed (container swelling) a hydrocarbon analyzer was used to 'snoop' the seals. Suspect containers were immersed in a water bath while under positive pressure to determine leak points. All leak points were repaired prior to any gravimetric analysis. During the diurnal

tests, all suspect containers were checked with the hydrocarbon analyzer and if necessary, repaired using the same methods.

Weight loss was used as the basis for determining relative permeation rates. Sealed containers were weighed using a high capacity balance (Sartorius Masterpro, 16k-gram capacity, sensitivity ± 0.1 gram) just prior to the start of each diurnal cycle. After each container was individually weighed and the weight recorded, they were placed in a Sealed Housing for Evaporative Determination (SHED) and exposed to a 1-day / 24-hour/ 1440-minute variable temperature profile (see Attachment 2). Containers were then post weighed after the 24-hour diurnal cycle and the weight loss calculated.

Cumulative weight loss by the containers as a function of time was plotted for all initial 24-hour test cycles. Data were considered acceptable when weight loss became linear with respect to time. All test data include the following information: calculated wetted surface area, weight lost per test (± 0.1 gram), and initial volume of test fuel. Container identification labels are described in Attachment 3.

Results

The average permeation rate from follow up tests of containers fluorinated at level 5 was determined to be 0.16 grams/gallon/day. This final rate is based on data averaged from tests of 6 containers and represents a total of 78 individual 24-hour diurnal cycles.

The average permeation rate from follow up tests of containers fluorinated at level 3 was determined to be 0.57 grams/gallon/day. This final rate is based on data averaged from tests of 6 containers and represents a total of 78 individual 24-hour diurnal cycles.

The average permeation rate from follow up tests of sulfonated containers was determined to be 0.07 grams/gallon/day. This rate is based on data averaged from tests of 8 containers and represents 104 individual 24-hour diurnal cycles

The following table compares the permeation results of the initial and secondary tests with those of the follow up tests. Please note that the average untreated HDPE container permeation rate of 1.57 grams/gallon/day is used as the baseline for determining the efficiency of the barrier surface treated containers.

Table 2

Initial Tests		Secondary Tests		Results	
Container Type	Average Rate (g/gal/day)	Secondary Tests (g/gal/day)	Follow Up Tests (g/gal/day)	Combined Average (g/gal/day)	Control Efficiency (%)
Fluorinated Level 5*	0.24*	0.43*	0.16	0.28	82.2
Fluorinated Level 3	0.42	0.93	0.57	0.64	59.0
Sulfonated**	**	N/A	0.07	0.07	95.5

* 2 sets of level 5 containers treated incorrectly and were removed from the data set

**Sulfonated containers treated incorrectly and were removed from the data set

It should be noted that not all data points recorded in the attached data sheets were used in determining the various average permeation rates. Several data points were not included in the overall calculations for reasons identified in the field data sheets. These include but are not limited to: balance errors, excessive weight loss due to incompletely cured sealant(s), and mechanical difficulties with the SHED.

Conclusions

Sulfonated containers when correctly barrier surface treated provide a substantial reduction in permeation rates as compared to untreated containers. Furthermore, the continued swelling and paneling of the container walls during the application of the variable temperature profile does not appear to degrade the barrier surface treatment. While this is in no way a substitute for a formal longevity study, the results are encouraging.

The fluorinated containers also provide a substantial reduction in permeation rates as compared to untreated containers. Staff chose to conduct the follow up tests of the fluorinated containers because in the initial test, two sets of level 3 fluorinated containers out performed two sets of level 5 fluorinated containers. The data from these tests clearly show that the two sets of level 5 fluorinated containers used in the initial test were incorrectly treated.

It should be noted that initial tests of fluorinated containers suggests that continued swelling and paneling of the container walls experienced during the variable temperature profile degrades the integrity of the barrier surface treatment. While a slight increase in overall rates can be seen in the attached data sets for the fluorinated containers, the follow up tests were concluded after completing only 14 consecutive diurnal cycles. Based on previous test results, staff would anticipate an increase in permeation rates from the fluorinated containers of approximately 50% in secondary tests.